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## REMOVAL OF METHYLENE BLUE FROM AQUEOUS SOLUTIONS USING RAW AND MODIFIED RICE HUSK

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**ABSTRACT**, one of the most important environmental pollutants is dye containing wastewaters. Methylene blue (MB) is a cationic dye that has carcinogenic and mutagenic effects on human. This study was investigated to remove methylene blue dye from aqueous solutions by raw and modified rice husk (Raw-RH and Modified-RH). The influence of various parameters including pH, contact time, adsorbent dose and initial dye concentration was studied on the dye removal efficiency in a batch system. The results showed that the equilibrium was obtained at the contact time of 90 min and the maximum dye removal was also occurred at pH 10 for both the Raw-RH and Modified-RH adsorbents. The experimental data were analyzed by the Langmuir and Freundlich isotherm models. The findings showed that the data were best fitted with the Freundlich isotherm. The obtained data for MB adsorption onto the Raw-RH and Modified-RH were also fitted via the pseudo-first order and pseudo-second order kinetic models. The correlation coefficients values ( $R^2$ ) showed that the adsorption kinetic described well by the pseudo-second order model. The results of this study indicated that rice husk can be used as an effective and low-cost adsorbent for the removal of methylene blue from aqueous solutions.

**Keywords:** Adsorption, Methylene blue, Rice husk, Aqueous solution.

### INTRODUCTION

Nowadays, the environmental pollution is considered as a global problem especially in developing countries (Nourmoradi et al. 2012). The textile wastewater is one of the most dangerous wastewaters that, due to its color, can lead to reduce the

permeability of light in water bodies and then make disorder to do photosynthesis process (Chojnacka 2006; Crini&Badot 2008). In the view point of aesthetics, these colored compounds affect negatively on the quality of drinkable water as well as some other uses (Radhika&Palanivelu 2006). Dye materials can cause allergy, dermatitis, skin rashes (Vijayaraghavan& Yun

2008), cancer (Pajootan et al. 2012) and genetic mutations in human (Ponnusami et al. 2007). Improper treatment and disposal of colored sewage resulted from textile, dying, printing and other related industries have led to make many environmental problems and issues around the world (Bayramoglu et al. 2007; Toor&Jin 2012). Methylene blue dye (MB) is a hetero-aromatic composition with a sap green color which is as powder form at the room temperature (Han et al. 2007). Many treatment methods including ozonation (Malik &Sanyal 2004), chemical oxidation (Koby et al. 2007), photo-catalytic and membrane processes (Lin et al. 2013; Shen et al. 2009) and biological procedures (Manu &Chaudhari 2003) are used to remove dye from textile wastewaters. However, some of these methods because of the costs of operation, high consumption of energy, producing much sludge and the time consuming are limited to use. Agricultural wastes are considered as an effective and low-cost absorbent to remove dyes from dye containing wastewaters. The low-cost agricultural waste materials including rice husk, banana peel, rape (Gong et al. 2008), sun flower stalks, volatile ash, charcoal, wheat and aerosphere chaff (Ghanizadeh&Asgari 2009) and red mud (Zazouli et al. 2013) are used to eliminate the organic and inorganic pollutants including dyes from wastewaters. Therefore, because of the plenty of agriculture wastes in Mazandaran province (Iran) and its effective adsorption property, it can be used to eliminate dye from wastewaters. The aim of this research was to use of raw and modified rice husk (Raw-RH and Modified-RH) for eliminating methylene blue (MB) dye from the aqueous environments. The effect of various parameters including pH, contact time, adsorbent dose and initial dye concentration was studied on the dye removal efficiency.

## 2. Materials and Methods

### 2.1. Materials:

Rice husk, as an adsorbent, was obtained from rice camlets of Mazandran province (Iran). Methylene blue (MB) dye was purchased from AlvanSabet Co (Iran). The chemical structure and characteristics of MB are presented in Fig 1 and Table 1, respectively. Other chemicals including citric acid, sulfuric acid and sodium hydroxide were provided from Merck Co (Germany).

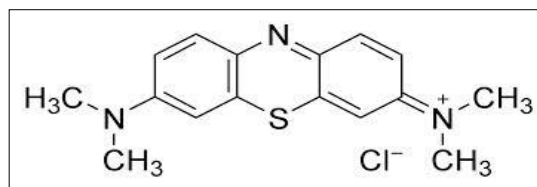


Fig. 1. Chemical structure of Methylene Blue (MB).

Table 1. General characteristics of Methylene Blue (MB)

Characteristic	MB
Generic name	Methylene Blue
Chemical formula	C <sub>16</sub> H <sub>18</sub> N <sub>3</sub> SCl
Molecular weight (g/mol)	373.90
$\lambda$ max (nm)	665
Appearance	Sap green powder

### 2.2. Adsorbent preparation

First, the adsorbent was washed by distilled water and then it was placed into a furnace for 2 hours at 90 °C. Thereafter, the adsorbent was mixed with citric acid (0.5mol/L) at a ratio of 1:12 for 30 minutes at room temperature (25 °C). Next, the sorbent was placed into the furnace at 50 °C for 24 hour. In order to implement the reaction between rice husk and acid, the adsorbent was introduced into the furnace at 120 °C for 90 minutes, then it was washed by distilled water and was filtered (fiber glass 0.25  $\mu$ m). The modified adsorbent was mixed in sodium hydroxide (0.1mol/L) with the proper ratio for 60 minutes. The sorbent was then washed by distilled water to eliminate the residual alkali, dried at 50 °C for 24 hour and ground (<250  $\mu$ m) for the subsequent use in the experiments (Chojnacka 2006; Li et al. 2008).

### 2.3. Adsorption experiments

The adsorption experiments were conducted in a batch system at room temperature (25°C) with 100 mL of the dye solution (10 mg/L) into a 250 mL Erlen Myer and agitated by an orbital shaker (150 rpm for 3 hr) at pH 10. After agitation, the suspensions were centrifuged (3600 rpm for 15 min) and the clear solution was analyzed for dye by an UV-Vis spectrophotometer (DR-5000 Hach) at maximum absorbance wavelength of 665 nm. All the experiments were conducted in duplicates and the mean values were applied. The removal efficiency and sorption capacity of the raw and modified rice husk (Raw-RH and Modified-RH) were determined by Eq. (1) and (2), respectively:

$$R(\%) = \frac{(C_0 - C_t)}{C_0} \times 100 \quad (1)$$

$$q_e = \frac{(C_0 - C_e)V}{m} \quad (2)$$

Where;  $R$  (%) and  $q_e$  (mg/g) are the removal efficiency and adsorption capacity, respectively.  $C_0$  (mg/L) is the initial dye concentration,  $C_e$  (mg/L) is dye concentration at the equilibrium,  $m$  (g) is the mass of the sorbent and  $V$  (L) is the volume of the dye solution (Faghihian et al. 2012).

### 2.3.1. Effect of contact time and pH

The influence of contact time on the dye removal was applied by 100 mL of the solution containing 0.1 g sorbent and 10 mg/L dye at various contact times (5-240 min) at pH 10. The pH experiments were also conducted at the optimum contact time with 0.1 g modified rice husk (Modified-RH) and 10 mg/L dye in the various pH values (2 to 10). The regulation of the solution pH was carried out through 0.1M  $H_2SO_4$  and 0.1M NaOH to the considered values. The contact time and pH samples were then centrifuged (3600 rpm for 15min) and determined for the dye concentration via the spectrophotometer.

### 2.3.2. Effect of adsorbate concentration and adsorbent dose

The effect of initial dye concentrations (10-200 mg/L) on the sorption was determined by 0.1 g Raw-RH and Modified-RH at the optimum contact time and pH at 25°C. The influence of adsorbent dose (2-25 g/L) on the adsorption was also done through 0.1 g adsorbent, 10 mg/L dye, optimum pH and contact time at 25°C. The suspensions were then analyzed the same as the above.

## 3. Results and Discussion

### 3.1. Effect of contact time

The results, Fig 2 (a), showed that the amount of methylene blue sorption was quickly increased over the first 45 min of the process and then was slowly increased as the time went forward up to 90 min for both the Raw-RH and Modified-RH. The equilibrium was acquired within 90 min. It may be due to the more availability of the sorption sites at the early step of the adsorption process and the adsorption sites were gradually occupied by the dye molecules as the contact time was increased (Faghihian et al. 2014). In this study, the amount of dye removal efficiency with the Raw-RH and Modified-RH was observed 83% and 96%, respectively. Shih (2012) reported that the

acid modification of rice husk reduced MB (50 mg/L dye) sorption efficiency from aqueous solution to 98% for CRH (clean rice husk), to 67% for NRH (nitric acid modified rice husk), to 59% for HRH (hydrochloric acid treated rice husk) and 55% for SRH (sulfuric acid treated rice husk) (Shih 2012).

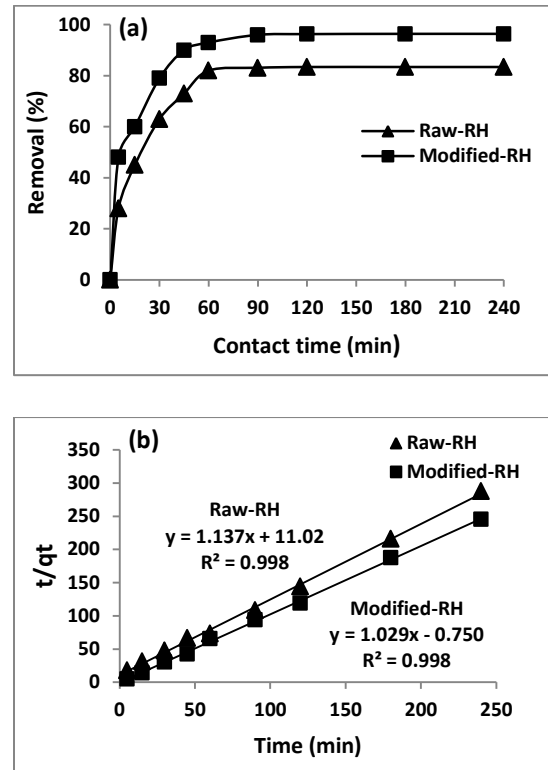


Fig. 2. (a) The effects of contact time on the removal of MB by the adsorbents (dye solution = 10 mg/L, initial pH =10 and sorbent conc. = 1g/L) and (b) pseudo-second order kinetic model.

### 3.3. Adsorption kinetics study

Adsorption kinetics study is helpful to predict the adsorption rate. Different sorption kinetic models including pseudo-first order and pseudo-second order kinetics were extensively applied to the adsorption data. The pseudo-first kinetic model is presented by Eq. (3):

$$\ln(q_e - q_t) = \ln q_e - k_1 t \quad (3)$$

Where;  $q_e$  and  $q_t$  (mg/g) are the quantity of adsorbed dye at equilibrium time and at the stated time, respectively.  $k_1$  is the specific rate constant for the first order kinetic model and  $t$  is the contact time.  $k_1$  and  $q_e$  are acquired from the slope and intercept of the linear plot of  $\ln(q_e - q_t)$  versus time ( $t$ ), respectively (Nourmoradi et al. 2013). The pseudo-second order kinetic model is also presented by Eq. (4):

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (4)$$

Where;  $k_2$  is the specific rate constant of the pseudo-second order kinetic model and other parameters are the same as the above.  $k_2$  and  $q_e$  are calculated from the intercept and slope through plotting  $t/q_e$  versus time ( $t$ ), as shown in Fig. 2 (b), respectively (Nourmoradi et al. 2013). The higher linear correlation coefficient of pseudo-second order kinetic model ( $R^2=0.998$  for both the sorbents) showed that this adsorption kinetic model fitted the sorption data better than pseudo-first order kinetic model. Shih (2012) and Ashiq et al. (2012) reported that MB sorption by acid and acid/base modified rice husk was in agreement with pseudo-second order kinetic model with higher correlation coefficient, respectively (Ashiq et al. 2012; Shih, 2012).

### 3.4. Effect of solution pH

Fig 3 shows the influence of solution pH on the adsorption of dye by rice husk. By increasing the initial solution pH, the dye removal efficiency was increased. As the solution pH was raised, the surface functional groups on the adsorbents were deprotonated and it changes to negatively charged surface (Teket et al. 2009). Otherwise, the MB dye molecules in the aqueous solution have the positively charged ions (Dogan et al. 2009). The increase of MB removal at higher pH may be due to the electrostatic attraction forces between the positive charged dye molecules and the negatively charged surface adsorbent (Chojnacka 2006; Ghanizadeh and Asgari 2009; Zazouli et al. 2013). The solution pH of 10 was the optimum condition to eliminate MB dye with both the Raw-RH and Modified-RH. At the initial pH of 10, the MB removal efficiency was found to be 80.9% and 93.8% for the raw and modified sorbents, respectively. Ashiq et al. (2012), Dogan et al. (2009) and Deng et al. (2009) reported that MB removal by various sorbents was increased with increasing solution pH (Ashiq et al. 2012; Deng et al. 2009; Dogan et al. 2009).

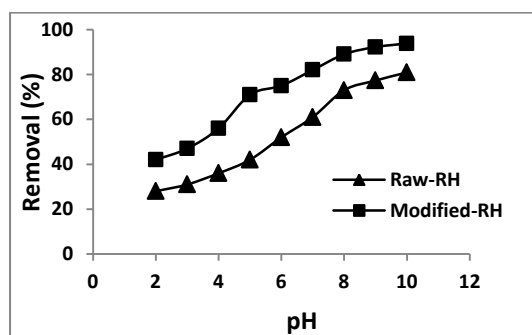


Fig. 3. The effect of pH on the removal of MB by the adsorbents (dye solution = 10 mg/L, contact time =90 min and sorbent conc. = 1g/L).

### 3.5. Effect of initial dye concentration

The effect of different dye concentrations on the adsorption was shown in Fig 4. As seen, the results showed that the dye removal efficiency was decreased from 83.0% to 25.0% and from 93.3% to 48.0% for Raw-RH and Modified-RH, respectively by increasing the dye concentration from 10 mg/L to 200 mg/L. An increase in initial dye concentration may be resulted in the decreasing dye uptake due to reduction in ratio of sorbent active surface to the pollutant molecules.

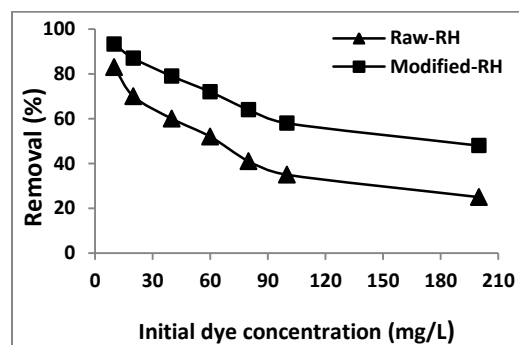


Fig. 4. The effect of MB concentration on the removal of MB by the adsorbents (contact time =90 min, pH=10 and sorbent conc. = 1g/L).

### 3.6. Adsorption isotherms study

The adsorption isotherms show the pollutant molecules distribution on the solid phase of sorbent at equilibrium state. Two adsorption isotherms including Langmuir and Freundlich were widely used for this purpose. The Langmuir sorption isotherm is based on the monolayer adsorption on the homogeneous surface of the adsorbent without interaction between the adsorbed molecules (Noorimotlagh et al. 2014). The Langmuir isotherm can be shown by Eq. (5):

$$\frac{C_e}{q_e} = \frac{1}{bQ_m} + \frac{C_e}{Q_m} \quad (5)$$

Where;  $C_e$  (mg/L) is the dye concentration at equilibrium time and  $b$  (L/g) is the Langmuir constant.  $q_e$  (mg/g) and  $Q_m$  (mg/g) are the sorption capacity of the adsorbent and the maximum adsorbent capacity at equilibrium, respectively.  $Q_m$  and  $b$  are achieved from the slope and intercept of plotting  $C_e/q_e$  versus  $C_e$ , respectively (Faghihian et al. 2014).

The Freundlich isotherm can be used for non-ideal adsorption on heterogeneous surface of the adsorbent that is described by Eq. (6).

$$\text{Log } q_e = \text{Log } K_f + \frac{1}{n} \text{Log } C_e \quad (6)$$

Where;  $K_f$  (L/mg) and  $n$  are the isotherm constants which show the capacity and intensity of the adsorption, respectively.  $K_f$  and  $n$  are determined from the intercept and slope of plotting  $\text{Log } q_e$  against  $\text{Log } C_e$ , respectively (Nourmoradi et al. 2014). The Langmuir and Freundlich isotherm parameters are presented in Table 2. As seen, the correlation coefficient of Freundlich isotherm ( $R^2 = 0.991\text{--}0.995$ ) for both the adsorbents was higher than Langmuir isotherm. The adsorption bond between adsorbent and adsorbate would be relatively strong if the value of  $n$ , acquired from the Freundlich isotherm, is more than one (Nourmoradi et al. 2013). Therefore, the  $n$  values of 1.47 and 2.91 obtained by this isotherm model showed that MB was properly adsorbed by Raw-RH and Modified-RH.

Table 2. Langmuir and Freundlich isotherm parameters for the dye sorption by rice husk.

Adsorbent	Langmuir isotherm			Freundlich isotherm		
	$Q_m(\text{mg/g})$	$b(\text{L/g})$	$R^2$	$K_f(\text{L/mg})$	$n$	$R^2$
Raw -RH	24.7	0.29	0.961	1.15	2.91	0.991
Modified-RH	21.4	0.31	0.971	1.10	1.47	0.995

### 3.7. Effect of adsorbents dose

Fig 5 shows the effect of adsorbent doses on the dye removal by raw and modified rice husk. As shown, with increasing the adsorbent dose from 2 g/L up to 10 g/L, the dye removal efficiency was increased from 54.0% to 82.7% and from 72.0% to 93.0% for Raw-RH and Modified-RH, respectively. But, the performance of both the sorbents was nearly constant at adsorbents doses more than 10 g/L. It may be due to the aggregating the active surface sites of the sorbent and as well as blocking the binding sites by the MB molecules that resulted in lower dye removal per unit sorbent. Therefore, 10 g/L of the adsorbent dose was adequate for the optimum removal of MB as shown in Fig 5.

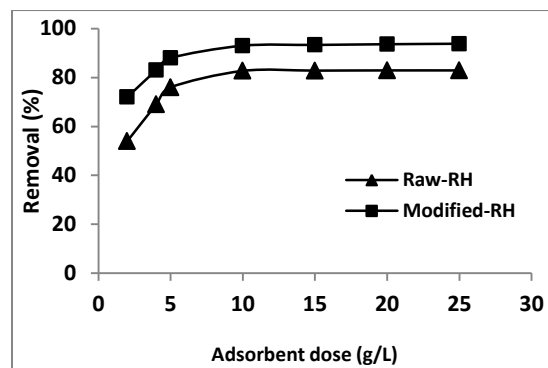


Fig. 4. The effect of adsorbent dose on the removal of MB (contact time =90 min, pH=10 and dye conc. = 10 mg/L).

### 4. Conclusion

In this study, surface modification of rice husk by citric acid was investigated for the removal of methylene blue (MB) from aqueous solutions. It was determined that the adsorption conditions were optimum at the contact time of 90 min and pH 10 for both Raw-RH and Modified-RH sorbents. The adsorption of MB onto the adsorbents was well described by the pseudo-second order kinetic model and Freundlich isotherm model. Because of the low cost, eco-friendly and non-toxicity of rice husk, this natural adsorbent can be considered as one of the best choices on the removal of MB from aqueous solutions.

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